



Method of Test for Cleanness Assessment of Metal Alloys

The Preparation of Button Specimens by Electron Beam Melting and the Characterisation of Recovered Inclusions.

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ABSTRACT

This report describes a method for the cleanness assessment of metal alloys in which a metal specimen is electron beam melted into a copper crucible to collect the non-metallic inclusions present in the specimen into a small area on the top surface of the cast sample known as a button. It also describes methods for the examination of the collected inclusions to assess the cleanness of the specimen material with respect to the number, size shape and approximate composition of the inclusions present.

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1 SCOPE

1.1 This test method describes procedures for melting metal specimens into a copper crucible using an electron beam to collect the non metallic inclusions, normally oxides, onto the metal top surface. Methods to analyse the collected inclusions are also described.

1.2 The method is often but not solely limited to alloys for which the non-metallic inclusions present in the alloy are too few for a statistically significant number to appear on a metallographic cross section.

1.3 The test has proved most useful for nickel-base superalloys with aluminium and or titanium contents which form stable oxides that are not reduced by carbon under the heating effect of the beam. The test can be used for some steels and cobalt alloys but the method should be verified for individual materials.

2 NORMATIVE REFERENCES

NOTE

A draft document:

"Test Method for Measuring Inclusion Content Using Analytical Skull Melting Part 1: Preparation of Buttons"

was prepared for ASTM Sub-committee E04: 09.07 for Standardising Test Method and Measurement, by R.C. McAllister.

Revision 1, dated May 15, 1986, of the ASTM document has been considered in the preparation of this present document. See also references 1 and 2.

3 DEFINITIONS

3.1 BUTTON (ALTERNATIVELY REFERRED TO AS ANALYTICAL SKULL)

A cast sample, normally a segment of a sphere, which has been electron beam melted and solidified under controlled conditions to concentrate the non metallic inclusions into a small area on the top surface of the sample.

3.2 CAP

The area on the top surface of a button, generally located at the centre, with a distinct solidification structure, containing inclusions and metal.

3.3 DRIP MELTING

A process in which a specimen is melted with an electron beam and the liquid is allowed to drip into a crucible.

3.4 OSCILLATION OF AN ELECTRON BEAM.

The movement of an electron beam in a straight line about a central point. A combination of oscillations in the x and y directions is used to control the beam so that it sweeps consistent and reproducible patterns.

3.5 STATIC DEFLECTIONS (SOMETIMES KNOWN AS RADIAL AND TANGENTIAL STATIC DEFLECTIONS)

The co-ordinates of the electron beam in the absence of oscillation.

3.6 RAFT (ALTERNATIVELY KNOWN AS THE INCLUSION CONCENTRATION AREA)

The area on the cap containing oxides, which may be agglomerated in one area. Sometimes the raft is not clearly discernible when viewed at low magnifications.

3.7 SPECIMEN (ALTERNATIVELY REFERRED TO AS AN ELECTRODE)

The material which is button melted. The specimen can take a variety of shapes.

4 SUMMARY OF METHOD

4.1 The objective of the test is to melt a metal specimen into a copper crucible using an electron beam and collect the non metallic inclusions, normally oxides, onto the metal top surface of the cast button for subsequent analysis.

4.2 A suitably prepared specimen is placed in a chamber, which is subsequently evacuated. Drip melting of the specimen is performed with an electron beam and the metal drops are collected in a clean, water cooled crucible (see 5.1) or hearth.

4.3 The inclusions are concentrated near the centre of the button's upper surface by the controlled action of the electron beam.

NOTE

It is considered that surface tension effects⁽³⁾ are mainly responsible for the entrapment of the inclusions on the surface of the metal, possibly assisted by a flotation mechanism⁽⁴⁾.

5 APPARATUS FOR ELECTRON BEAM MELTING

5.1 The apparatus consists of:

- a vacuum chamber;
- suitable vacuum pumps and valves;
- sufficient pressure gauges connected to the chamber to monitor the pressure in the range of 1 bar to $\sim 10^{-6}$ mbar;
- an electron gun with sufficient beam power to melt the specimen (a power of about 20kW is sufficient);
- power and positional controls for the electron beam;
- means to rotate and translate the specimen in a controlled way;
- a water cooled copper crucible with a sphere segment shape.

- 5.2 The pumping system shall be able to achieve a pressure of at least 4×10^{-4} mbar for button preparation.
- 5.3 The cold leak-up rate of the chamber (see NOTE 1) shall not exceed 3×10^{-3} mbar l s^{-1} . If it is not possible to measure the cold leak-up rate (i.e. no high vacuum valve is fitted), then an alternative method agreed with the customer shall be employed.
- 5.4 The copper crucible (see 5.1) shall be water cooled. Crucibles with a diameter of about 40 mm are commonly used. This does not preclude other sizes of crucible (see NOTE 2) provided the size is documented in the results.
- 5.5 The controls of the electron beam shall at a minimum allow:
- 5.5.1 Sufficient static deflection in the x and y directions to allow the heating and melting of the specimen and controlled solidification of the button.
 - 5.5.2 A method to control the oscillations of the beam to vary circular beam sweep with a maximum diameter corresponding to the diameter of the crucible.
 - 5.5.3 A method to focus and defocus the electron beam. The minimum diameter of the electron beam when focused shall be 10 mm, when viewed with a fluorescent screen.
 - 5.5.4 A method to control the power of the electron beam by varying the accelerating voltage or the emission current (see NOTE 3).
- 5.6 There shall be provision for specimen movement such that:
- 5.6.1 The specimen can be rotated to allow it to be melted symmetrically (see NOTE 4).
 - 5.6.2 The specimen can be raised and lowered at a reproducible speed (see NOTE 4).
- 5.7 Manual, microprocessor or microcomputer methods are used to control the melting process. The latter two methods are to be preferred providing improved reproducibility in controlling the test parameters.
- 5.8 An observation window which absorbs X-rays shall be provided to enable a clear view of the process.
- 5.9 The X-ray count generated by the electron beam at typical power with a typical specimen shall be checked with an X-ray monitor at regular intervals around all likely sources of X-ray leakage such as seals and windows. This is especially important when any changes are made to equipment such as renewing seals. The measured values shall not exceed the exposure to X-rays recommended in the local health and safety regulations. The results of all X-ray tests shall be recorded.

NOTE 1

A stainless steel chamber is recommended and precautions shall be taken with a mild steel chamber to maintain it rust free by regular cleaning and pumping down when not in use.

Also a low humidity environment is recommended to prevent rusting of the surface of a mild steel chamber.

NOTE 2

With larger crucibles the power levels required to maintain the metal surface molten are high resulting in an increased tendency for the inclusions to melt⁽⁵⁾.

NOTE 3

The use of an ammeter and a voltmeter, suitably calibrated, to indicate beam current and accelerating voltage is recommended

NOTE 4

It is advantageous for the specimen rotation to be continuously variable in the speed range of ~0.2 to 10 revolutions per minute, and the speed of raising and lowering to be controlled in the range 2 mm min⁻¹ to 200 mm min⁻¹. The use of a suitably calibrated meter is recommended to indicate the vertical movement of the specimen.

6 CALIBRATION OF APPARATUS

6.1 The position of the electron beam shall be determined with a metal plate coated with a material which fluoresces under an electron beam at low power.

6.2 The focus and static deflection positions of the beam (see NOTE 1 and figure 1) shall be determined with respect to the control settings in manual and automatic control at low power. It is convenient to establish the control settings corresponding to the beam conditions for:

- impingement at the centre of the bottom surface of the crucible;
- impingement at the centre of the top surface of the button;
- impingement at the bottom of the specimen at the start of the melt;
- impingement at the top of the specimen at the start of the melt;
- the fully focused beam impinging at the top surface of the button;
- the defocused beam used in the melting program.

6.3 The controls for the oscillation of the beam shall be adjusted so that the beam describes a circle in a plane corresponding to the top surface of the button and the control settings required to vary the diameter of the circle shall be determined.

6.4 The control settings for the electron beam established in clauses 6.2 and 6.3 shall be checked at typical melting power levels with a specimen and button in place.

6.5 The speed of the lowering and raising of the specimen shall be determined in units of mm min⁻¹. If the speed is continuously adjustable the control settings shall be checked against the speed at selected intervals. If a meter indicating the velocity of the specimen is fitted the meter shall be calibrated.

6.6 The direction of rotation of the specimen is generally fixed so that the hot specimen is easily viewed and the speed shall be determined in revolutions per minute. If the speed

of rotation is continuously variable the control settings shall be checked against the speed at selected intervals.

6.7 The accelerating voltage of the electron beam (see NOTE 2) shall be checked on installation of the apparatus either at the normal working voltage or within the voltage range used. The voltmeter indicating the accelerating voltage shall be calibrated.

6.8 The emission current drawn by the gun shall be checked on installation within the normal operating range. The ammeter indicating the emission current shall be calibrated.

NOTE 1

It is recommended that the noise levels on the voltages controlling the coils are checked with a suitable instrument since small unwanted voltages can affect the focusing and positioning of the beam (a typical acceptable noise level is 50 mV peak to peak).

NOTE 2

The high voltages used to accelerate the electrons are lethal and care must be exercised in any measurements or adjustments of parts of the apparatus in contact with high voltages.

7 SAMPLING AND TEST SPECIMENS

7.1 A wide variety of starting materials produced by a number of manufacturing processes is likely to be of interest so the sampling and any machining plans shall be agreed with the customer before any specimen preparation is performed.

7.2 The shape and size of the specimen (see NOTE 1) shall be agreed with the customer and this geometry shall be maintained to allow direct comparisons of the results.

7.3 For large ingots such as those produced by VIM, VAR, ESR (EFR), EBCHR and PCHR (see Appendix) the exact position of the specimen within the cast ingot shall be specified.

7.4 The diameters of ingots of cast alloys are generally less than those types specified in NOTE 1 and it is sometimes possible to melt the ingots directly. If specimens are prepared the exact position of the specimen within the cast ingot shall be specified.

7.5 The melting of irregular shapes (for example turbine blades) is allowed provided a reproducible process is used for an identical shape.

7.6 When specimens are prepared from starting material, the preferred rough cutting methods are electrical discharge machining and cutting with tungsten carbide or silicon carbide abrasive wheels. Alumina abrasive wheels shall not be used since oxide particles can be entrapped in the metal and subsequently interfere with the test.

7.7 Final cutting shall be performed with tungsten carbide or silicon carbide tools. There shall be no discolouration of the metal caused by the cutting.

7.8 Specimens shall be thoroughly degreased with a suitable solvent (see NOTE 2), preferably in an ultrasonic cleaner.

7.9 If obvious cracks or porosity are observed in the specimen these shall be reported to the customer.

NOTE 1

A convenient specimen size and shape is a cylinder 35 mm in diameter by 90-100 mm in length giving a melted button mass of about 700g for a nickel-base superalloy.

NOTE 2

Acid cleaning or electropolishing is not generally recommended since an oxide film may be formed on the surface.

8 PREPARATION OF APPARATUS FOR TEST

8.1 The vacuum chamber shall be thoroughly cleaned to remove any deposited metal vapour from the internal surfaces and particulate material which may be present.

8.2 The internal surface of the crucible shall be wire brushed to a bright shiny appearance. It shall be finally cleaned using a solvent impregnated cloth or tissue followed by the removal of all particulate material, dust etc., using a vacuum cleaner. If the crucible has become badly scored or grooved it shall be repaired or replaced.

8.3 A support rod shall be attached to the specimen. It is preferable but not essential that the support rod is made from the same alloy and cast as the specimen. Support rods are typically round bar with a diameter of 8-10 mm and length of about 95 mm. They are normally attached to the specimen by electron beam melting in the vacuum chamber and in such a way as to allow the specimen to rotate concentrically during button preparation.

8.4 The support rod shall be clamped in its holder such that the specimen revolves concentrically. The height of the specimen above the crucible is an important parameter in the test (see NOTE).

8.5 The vacuum chamber shall be evacuated to a pressure $< 4 \times 10^{-4}$ mbar. The test can be conducted under a maximum inert gas back pressure of 1×10^{-2} mbar to reduce the likelihood of oxide inclusion decomposition during the melting of the button.

NOTE

Sintering and/or melting of oxide inclusions may occur when they are under the electron beam, particularly when it is at high power and focused to a small spot. It is, therefore, recommended that the specimen height above the crucible is set such that during melting when the beam is focused on the specimen it does not impinge on the button surface.

9 PROCEDURE

9.1 The following basic procedure^(4,6-12) shall be followed in conducting tests to ensure optimum collection efficiency and stability of inclusions during the test.

9.2 The test shall be performed at as low a temperature as possible by maintaining low beam power to minimise the sintering and/or melting of oxide inclusions during the test.

9.3 The specimen shall be rotated during the heating and melting stage at a speed (see NOTE 1) which in combination with the beam power input will ensure that it is uniformly heated and melted.

9.4 The test shall include the following stages:

9.4.1 Outgassing of crucible

It is important to ensure that all occluded or adsorbed gas is removed from the crucible surface prior to melting. This can be achieved by the use of a low power diffused oscillating beam to heat the internal crucible surface.

9.4.2 Heating of specimen

The rotating specimen shall be heated by a defocused electron beam from the bottom to a position near the top. The maximum temperature achieved shall be close to the melting point of the specimen and care shall be taken to avoid melting the support rod. Heating can be achieved either by:

moving the beam with the specimen fixed;
or moving the specimen vertically through the electron beam in a fixed position.

9.4.3 Formation of tip on specimen

A focused beam of sufficient power is used to melt the bottom of the rotating specimen to form a "pencil point" to enable the specimen to melt by a drip melting process.

9.4.4 Drip melting

The rotating specimen shall be drip-melted into the copper crucible to form a button sample. The shape of the specimen tip shall be controlled to maintain drip melting at a near constant rate and avoid any solid pieces falling into the crucible. In addition to melting the specimen, the beam shall also be programmed to maintain a liquid layer on the surface of the button throughout the melting stage at a temperature slightly above the melting point of the specimen (see NOTE 2).

The support rod shall not be melted.

At the end of the melting stage any remaining specimen shall be immediately raised out of the path of the electron beam.

9.4.5 Controlled solidification

The surface of the button shall be solidified in a controlled manner such that solidification starts at the outside circumference and the inclusions are not trapped in solid metal but remain on the liquid surface. The aim shall be to collect the inclusions within a cap near the centre of the top button surface (see NOTE 3).

9.5 To avoid oxidation of the button, it shall be allowed sufficient time to cool (typically 30 minutes for a nickel-base superalloy), before breaking the vacuum and removing the button from the chamber.

9.6 Care shall be taken to prevent contamination of the button by touching or scraping the cap surface.

9.7 The button shall be clearly identified either by writing on the bottom surface and/or clearly identifying the container in which it is stored (see NOTE 4).

9.8 In the event of any of the following occurrences a test shall be invalid:

9.8.1 The electron beam is interrupted for longer than 5 seconds during any part of the melting and solidification process.

9.8.2 Unmelted material falls from the specimen.

9.8.3 Buttons are produced by non-standard, non-established practices such as failure of the control system or operator error.

9.9 A log of each melt shall be kept and the following process variables shall be recorded:

beam power as a function of time.

beam oscillations and static deflections as a function of time.

rate of sample advance and rotation.

beam interruptions.

height of specimen above crucible.

dimensions of crucible and water flow rate.

NOTE 1

For furnaces where the speed of rotation of the specimen can be varied, it may be advisable that the speed is varied between the heating and melting stages. Also the larger the diameter the lower the speed of rotation required for the same power input.

NOTE 2

When the facility for oscillating the beam between two positions over a short time period (typically 0.5 to 1.5 sec) exists the preferred method of melting is to separate the melting of the specimen from the maintenance of the liquid pool on the button surface. Then, ideally, the beam can be focused on the specimen and de-focused on the button surface, transcribing a circular pattern following the circumference of the button outwards as melting proceeds.

NOTE 3

Controlled solidification can be achieved by simultaneously programming the beam to move in a circular pattern from the circumference to the centre of the button surface and reducing the beam power to zero. It is recommended that the process is programmed such that the end corresponds closely to the final solidification of the surface of the button.

NOTE 4

The button can be stored in a cylindrical "lozenge" tin provided it is protected from contact with the internal surface of the tin by placing in a polythene bag. Packing should be used to prevent movement of the button in the tin during transport.

10 EXAMINATION OF BUTTONS AND CLEANNES MEASUREMENTS

10.1 The exact form of the examination of the buttons to obtain a cleanness rating shall be agreed with the customer.

10.2 The examination methods⁽¹³⁻¹⁶⁾ may be conveniently sub-divided as follows into:

- preliminary;
- optical;
- scanning electron microscopy;
- others including extraction of the inclusions.

10.3 Preliminary examination.

10.3.1 The surface of the button shall be visually examined for signs of oxidation and if excessive the test shall be rejected.

10.3.2 The lower surface of the button shall be examined for surface laps which are permitted if the lapping is regular (the result of breaking the meniscus between the molten metal and the copper crucible); laps shall be no deeper than 7 mm and there shall be no evidence of trapped oxide particles when the surface is examined at a magnification of approximately X10.

10.3.3 The mass of the button shall be recorded.

10.4 Optical examination.

10.4.1 The button cap shall be examined optically and the size and shape of the cap observed (see NOTE).

10.4.2 The cap shall be examined optically at higher magnifications and regions containing oxide inclusions observed (see NOTE), in particular the distribution of the inclusions within the cap and whether there are indications of melting and/or sintering of the inclusions. Any inclusions observed outside the cap area shall be noted.

10.5 SEM examination

10.5.1 In order to clearly resolve small inclusions, the specimens shall be examined in the scanning electron microscope (SEM). The contrast modes which may be employed are:

- secondary electron;
- back scattered electron;
- cathodoluminescence.

In addition inclusions can be chemically analysed in the SEM using energy dispersive X-ray analysis.

10.5.2 A list of common phases and artifacts associated with the examination of phases is included in table 1.

10.5.3 The most useful contrast mode for routine analysis is the back-scattered electron image since the majority of buttons contain alumina based inclusions which appear dark against the lighter matrix. Pores also appear dark and can be distinguished by their low overall X-ray count rate and in the secondary electron contrast mode in which pores also appear dark but inclusions appear grey. The cathodoluminescence image is unreliable for distinguishing the alumina since the intensity is critically dependent on the amount and type of impurity associated with the alumina, but the signal is useful in finding areas of oxide.

10.5.4 For the examination of buttons containing zirconia based inclusions the contrast between the inclusion and the matrix is insufficient for identification in the back scattered mode. Either the energy of the X-ray peaks or in some cases the cathodoluminescence image can be used.

10.5.5 Hafnia is frequently found in alloys which contain hafnium. It is characterised by an intense cathodoluminescence signal and in the back scattered electron image it appears bright against the dark matrix. In the back scattered electron mode care shall be taken to distinguish the hafnia from refractory metal carbides which also appear bright.

10.5.6 Any indications of sintering or melting of inclusions shall be noted.

10.5.7 The determination of the size, shape and approximate composition of the inclusions can be determined by a variety of standard methods which can include automatic methods with suitably equipped microscopes. Particular care shall be taken in distinguishing the boundaries of the particles. In some cases it will prove impractical to automatically delineate the particle boundaries preventing the use of automatic methods for the separation of individual particles.

10.5.8 If quantitative chemical analysis of the particles by energy dispersive X-ray methods is to be attempted a correction programme which is applicable for the chemical analysis of rough surfaces shall be applied.

10.5.9 From the total projected area of the inclusions the NORA index can be determined (see NOTE).

10.6 Other examination methods

10.6.1 Amongst other methods for characterising the inclusions on the cap is one where the inclusions are isolated by chemically or electrolytically dissolving the metal matrix. The extracted inclusions are weighed and the ratio of the mass of inclusions to the total mass of the button is a measure of the cleanness of the specimen. This measure is less sensitive to the effects of sintering and melting the particles and is more difficult to perform than the NORA index (see NOTE).

NOTE

The button cap and the regions containing oxides may be photographed and the projected areas, maximum dimensions and an indication of shape of the larger inclusions may be measured by an appropriate method. If the majority of the inclusions can be resolved the total projected area of the inclusions may be determined and the NORA Index (Normalised Oxide Raft Area) may be calculated on the basis:

NORA Index = total projected area of oxide inclusions/ mass of the button.

The units commonly used are $\text{cm}^2\text{kg}^{-1}$ or $\text{mm}^2\text{kg}^{-1}$.

11 INTERFERENCES AND UNCERTAINTIES

11.1 The direction of the liquid flow for some alloys is dependent on the concentration of surface active elements such as sulphur⁽³⁾. At low concentrations (below ~10 ppm of sulphur in the nickel-based alloy IN718) the liquid flows from the hot to the cold part of the pool and this has the potential of trapping inclusions at the edge of the button. The problem can be overcome by adding nickel sulphide to the melt or careful programming of the position of the electron beam.

11.2 Some elements present at low levels in the base alloy can interact with the inclusions lowering their melting point. An example is yttrium⁽⁵⁾ which reacts with alumina such that the inclusions melt during the button test. It should be noted that these elements are sometimes present at concentrations below the normal reporting levels in the bulk composition.

11.3 If inclusions melt they can increase in surface area and thus the projected total inclusion area is larger than for the unmelted inclusions, with a correspondingly larger NORA index (see NOTE to clause 10). In this case, provided that the inclusions are of similar composition in two casts of the same alloy it is possible to compare their cleanliness but not evaluate the absolute size of the inclusions. In order to avoid the melting of inclusions the melting programme should be performed at as a low a temperature as possible⁽⁵⁾.

11.4 The test is unsuitable for inclusions which are wet by the liquid metal and therefore do not collect on the button surface.

11.5 The mass of material to be melted to obtain a result within a certain confidence limit is dependent on the volume fraction, size and distribution of the inclusions⁽¹⁷⁾. Guidance on the volume melted to obtain an 80% confidence of locating inclusions of certain sizes is given in figure 2 assuming that inclusions are spheres and are homogeneously distributed in the material. The left hand part of the figure shows the area that needs to be examined to obtain an 80% confidence of locating inclusions.

11.6 The variance of the test method using the NORA index⁽¹⁸⁾ has been estimated by melting buttons from adjacent specimens. Duplicate solid specimens were obtained from the bottom of a cast bar by electro discharging two cylinders axisymmetrically. Twelve such duplicate specimens were melted, and the average for the parameter $3\sigma/x$ was 0.19, where x was the average NORA index and σ the standard deviation (see table 2).

11.7 Experiments involving the incorporation of inclusions into powder metal samples and determining the efficiency of inclusion recovery have been performed by various workers⁽¹⁹⁻²²⁾. For high concentrations (~50 ppm) of alumina type inclusions with a diameter of 100 μm the collection efficiency was about 90%. At lower concentrations the results were more difficult to interpret although one group using spinel particles (100 particles/kg) in the 45-50 μm size range claimed a recovery rate of 80% with the alloy N18 but only 40% with IN718 (see also 11.1 and 11.2). Table 3 compares the results from doping experiments on the efficiency of inclusion collection.

11.8 Preliminary experiments⁽²³⁾ involving the direct incorporation of a known number of spinel particles into a specimen showed collection efficiencies varying between 70% to 100% for particles in the size range 50 to 100 μm . Some sintering of the particles was observed when the electron beam heated the pool during the time it was melting the specimen (see NOTE to clause 8).

11.9 Round robin exercises⁽²⁴⁾ have shown that there can be large variations in the results amongst the participants although there is agreement in the ranking for individual casts (see table 4 and figure 3). A possible explanation is that the beam powers used by the different participants differ very significantly. At the higher power levels there is a tendency for inclusions to melt affecting the measurement of cleanness.

12 REPORT

The report of the test shall contain the following minimum information:

- (a) name of the testing laboratory;
- (b) unique identification of report;
- (c) name and address of client;
- (d) details of test material, including material type, manufacturing code, batch number, dimensions and shape;
- (e) date of receipt of material and test;
- (f) reference to this method of test;
- (g) the height of the specimen above the top surface of the crucible at the beginning of the melting sequence;
- (h) dimensions of the crucible and water flow rate;
- (i) sufficient information about the melting procedure such that the furnace controls could be reprogrammed to reproduce the melting cycle, for example: the voltage; the current; the static deflections and oscillation of the electron beam; the raising and lowering speeds and the rotation speed of the specimen at particular times in the procedure;
- (j) any peculiarities during melting such as arcing of the electron gun;
- (k) identification of video recording where applicable;
- (l) method(s) employed to characterise inclusions, ie optical, scanning electron microscope or extraction technique;
- (j) contrast mode(s) for methods using the scanning electron microscope;
- (k) technique employed for calibration of linear dimensions and chemical analysis;
- (l) copies of micrographs used in establishing cleanness level with qualitative chemical analysis if performed;
- (m) agreed measure of cleanness such as projected area of a fixed number of largest particles; the NORA index; the ratio of mass of extracted inclusions to mass of button or any other agreed measure;
- (n) remarks on general appearance of the inclusions such as evidence for sintering or melting;
- (j) signature of person(s) responsible for test and authorising issue of report.

NOTE: For routine presentation of results a standardised format can be usefully adopted.

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APPENDIX

ABBREVIATIONS NORMALLY USED FOR INDUSTRIAL MELTING PROCESSES

VIM	Vacuum Induction Melting.
VAR	Vacuum Arc Remelting.
ESR(EFR)	Electro Slag Remelting (Electro Flux Remelting).
EBCHR	Electron Beam Cold Hearth Refining.
PCHR	Plasma Cold Hearth Refining.

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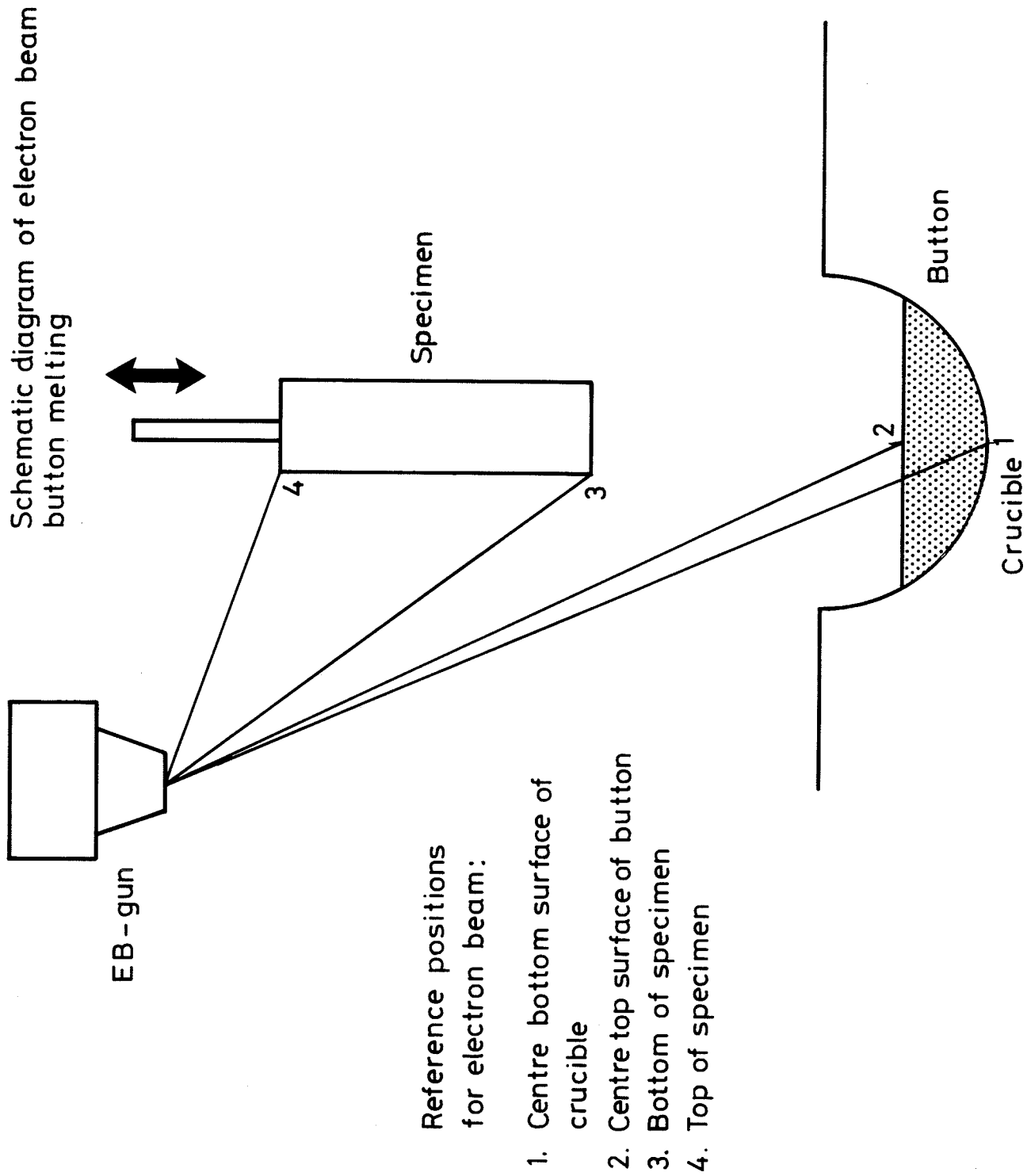


FIGURE 1

SURFACE AREA AND VOLUME REQUIRED TO BE EXAMINED TO OBTAIN A RELATIVE ERROR OF 20% IN MEASURING
VOLUME FRACTION FOR DIFFERENT DIAMETER INCLUSIONS⁽¹⁷⁾

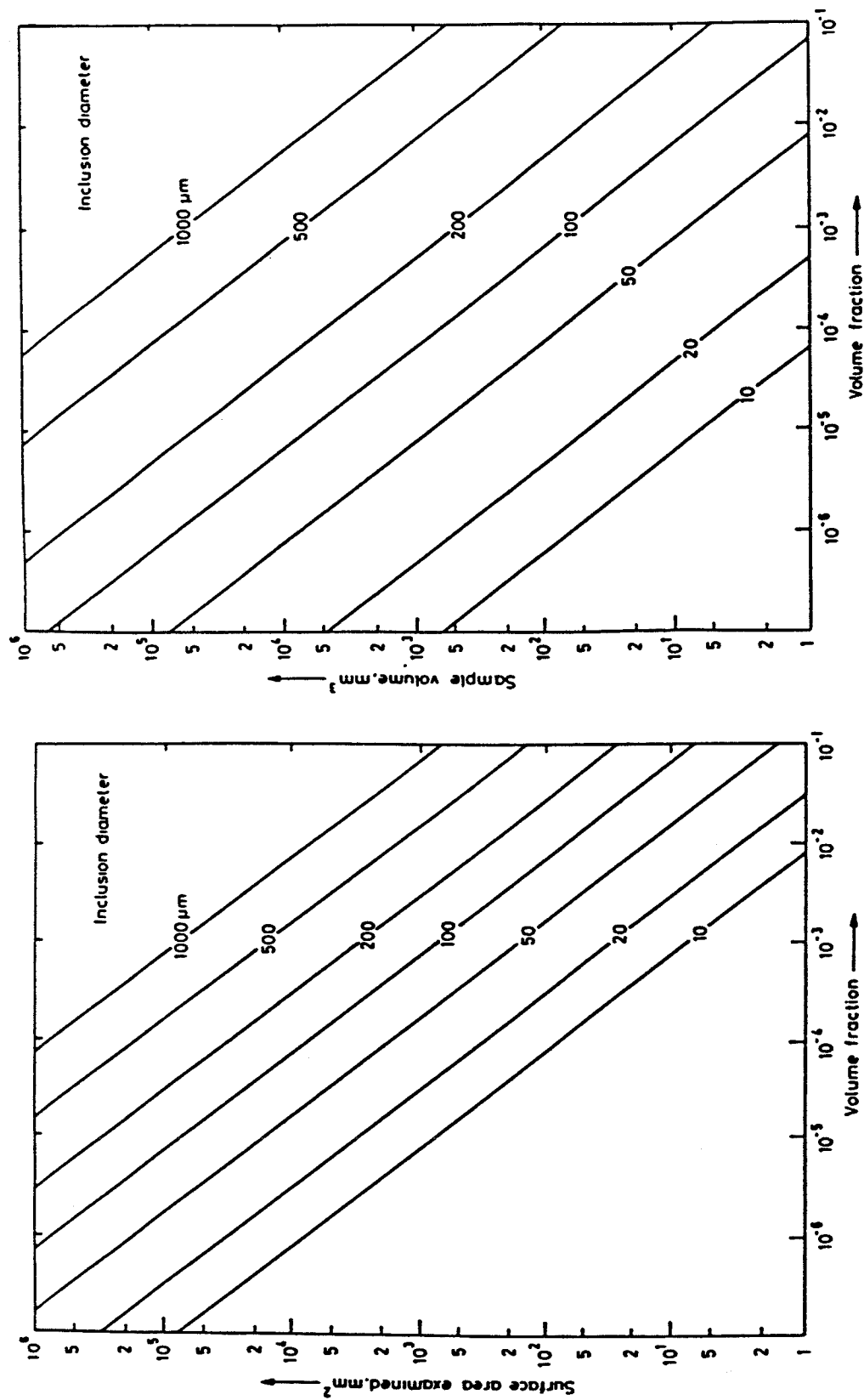


FIGURE 2

SUMMARY OF CONTRASTING MODES IN THE SEM⁽¹¹⁾

Inclusion type	Inclusion response		Comments
	Cathodoluminescence (CL)	Back scattered electron (BSE)	
Al ₂ O ₃	Intensity variable dependent on composition; normally red, low intensity; Cr ³⁺ gives "smeared image"	Darker than Ni matrix	BSE good for Al ₂ O ₃ in cast stock. Pores a problem with powder product * SE or X-ray helps.
ZrO ₂	Blue reported in literature; high intensity	Same contrast as Ni matrix	Bright CL image reveals ZrO ₂ - X-ray helps.
HfO ₂	Blue/white observed; high intensity Al content reduces intensity	Brighter than matrix	{ { { { { Use CL and BSE to separate HfO ₂ from carbides
HfC or other carbides	-	Brighter than matrix	
TiN	-	Darker than matrix	Generally very small
Pores	-	Darker than matrix	* SE image X-ray image

* SE = Secondary electron

TABLE 1

DUPLICATE "BUTTON" RESULTS⁽¹⁸⁾

Average cm ² /kg	Standard Deviation	3σ/x
0.615	0.025	0.12
0.395	0.045	0.34
0.205	0.025	0.36
0.365	0.005	0.04
0.17	0.00	0.00
0.225	0.025	0.33
0.10	0.01	0.3
0.77	0.03	0.12
0.635	0.025	0.12
0.67	0.02	0.09
0.90	0.06	0.2
0.82	0.06	0.22

Average for N = 12 is 0.19

TABLE 2

COMPARISON OF EFFICIENCY STUDIES FOR EBBM

Concentration Level	Dopant Types	Size/Size range	Material	Analysis Method	Efficiency	References
$\sim 10^5$ particles per kg	Al_2O_3	100 μm 50 μm 40 μm	Rene 95	Extraction	100% (for 100 μm size) to 30% (for 40 μm size)	4 (at GE)
20-100 particles per kg	Al_2O_3 SiO_2 MgO	80 μm to 500 μm	IN718	SEM	Difficult to interpret	20 (at GE)
	Al_2O_3	80 μm to 500 μm	Rene 95	SEM	As above	
$\sim 10^5$ particles/kg $\sim 10^3$ particles/kg $\sim 10^2$ particles/kg	Al_2O_3	50 μm	API	SEM	Better than 90% for 10^5 /kg concentration γ interfered with recovery experiment at lower concentrations	19 (at NPL)
100 particles/kg	Al_2O_3 -MgO Al_2O_3 -MgO Al_2O_3 -MgO	100-125 μm 45-50 μm 45-50 μm	N18 Astroloy IN718	SEM SEM SEM	- Good Recovery - better than 80% - 40% recovery	21,22 (in France)

TABLE 3

PARTICIPANTS IN THE ROUND ROBIN EXERCISE ORGANISED BY PROFESSOR TIEN

Company	Manufacturer of Furnace + Control System		Analytical Method	1st Round Robin	2nd Round Robin
Teledyne-Allvac	Leybold	Microprocessor	Optical	✓	
Carpenter Technology Corporation	North-Hill	Computer	SEM	✓	
Howmet Corporation	Leybold	Microprocessor	Optical (SEM)	✓	✓
SNECMA, France	Leybold	Computer	SEM		
Nippon Mining Company, Japan	Leybold (possibly modified)	Computer	SEM	✓	✓
University of British Columbia, Canada	Laboratory	Manual	-		
Special Metals Corporation	Laboratory	Manual	Optical		✓
National Physical Laboratory, UK	Leybold	Computer	(Optical) SEM		✓
GE Aircraft Engines	See Teledyne-Allvac, Carpenter, Wyman -Gordon		SEM		
INCO Alloys Ltd, UK	See NPL		SEM		
(Wyman-Gordon)	North-Hill	Computer	Optical SEM		

TABLE 4

Results of laboratory intercomparison
for EBBM test co-ordinated by
Professor Tien

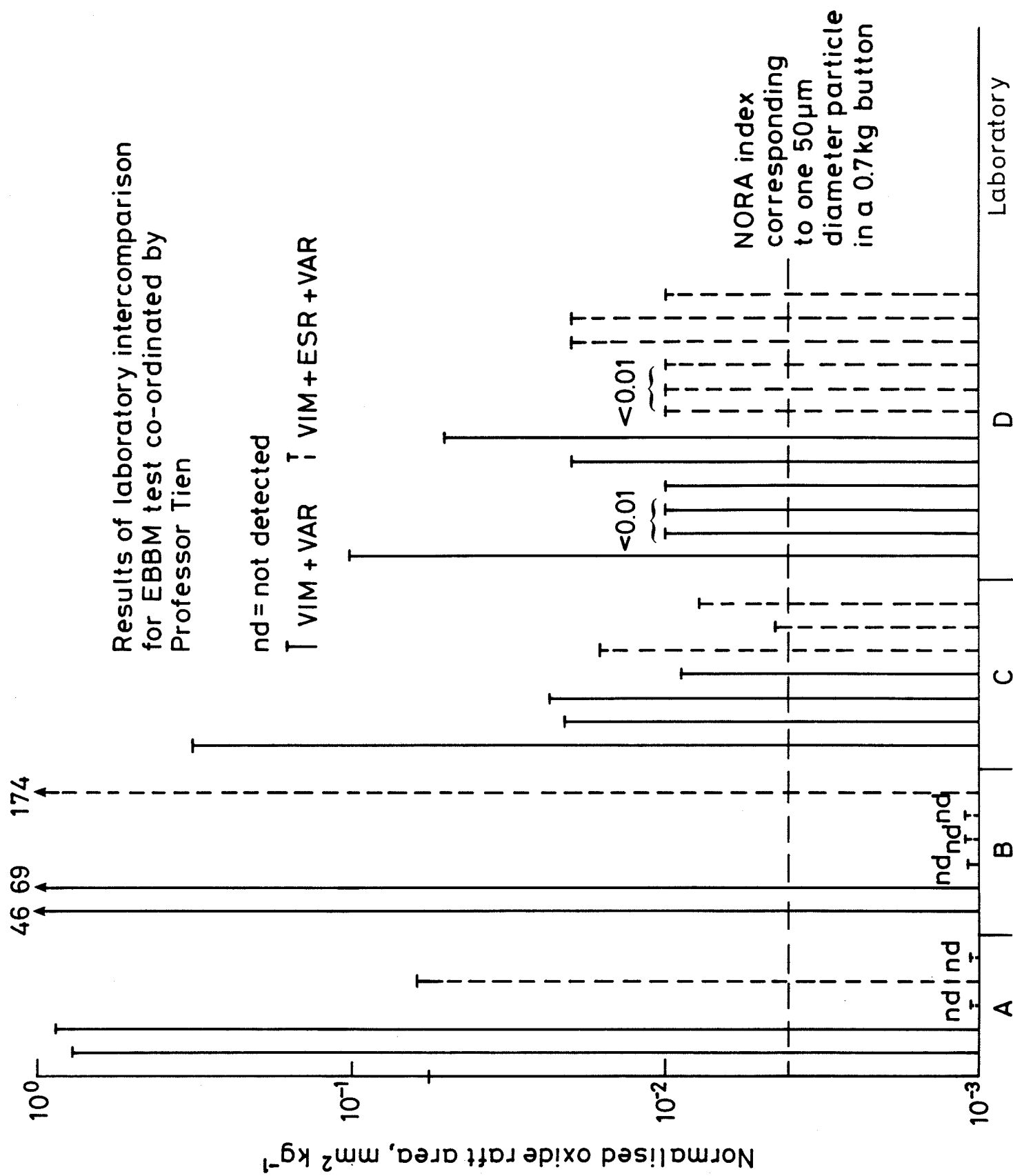


FIGURE 3